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Synthesis and Characterization of Metallic Bivalent and Trivalent Aerogels by Epoxide and Alkoxide Addition Method

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Aerogels are materials used as targets for laser experiences. These materials are high surface materials and low density. To elaborate aerogels, the sol-gel method is mainly used for high valence element (>2). But this method is note appropriate for elaborate low valence like CuO. Nevertheless, thanks to a complexant agent such as a polymeric acid [1-2], a three-dimensional structure is obtained and finally a gelation occurs. After a drying in CO_2 supercritical conditions, an aerogel is synthesized. The drying allows replacing the solvent located in the pores by air keeping initial structure. In this study, the epoxide and alkoxide addition method is described. The alkoxide method is known to be more expansive synthesis than epoxide method, in view of alkoxide precursor use (frequently unavailable, difficult to obtain). The protocol of synthesis is optimized according to molar ratios (H_2O/precursor; epoxide/precursor; complexant/precursor), type of epoxide, type of solvent, the nature of inorganic salt or metal alkoxides (= precursor according to addition method) and more precisely the counter anion, hydrolysis time and the gel time. Tests of synthesis were carried out on bivalent (Cu^{2+}) and trivalent (Yb^{3+}, Er^{3+}, Ce^{3+}) elements. Different cycles of drying are tested in order to bring about some change in the structure and the texture of aerogel. These materials are characterized using elemental analysis, nitrogen adsorption/desorption analysis, microscopy and pycnometry. To understand the mechanism of gelation via the epoxide and alkoxide addition, difference of reactivity of some parameters is discussed. Three mainly steps of synthesis (hydrolysis, complexation and condensation) were analysed by nuclear magnetic resonance and infrared spectroscopy in order to dry to determine the formed molecule structure.

References

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